Section 5: Identification of Amphetamines and Methamphetamines

I. Introduction:

Amphetamines, methamphetamines and related Phenethylamines are screened and analyzed by GC/FID and subsequently confirmed by GC/MS. However, a preliminary analysis is performed using the computerized Identidex Imprint Identification program within the Micromedex Healthcare Series. The samples are extracted by a simple solvent extraction procedure. However, an extraction procedure specific for non-volatile organic poisons can also be used (See Section IX).

II. Reagents:

A.) Petroleum ether
B.) Ammonium hydroxide (NH_4OH)
C.) Methanol (GC solvent rinse)
D.) 9:1 Methylene Chloride/NH_4OH

III. Equipment:

A.) Analytical balance
B.) Magnifying microscope or magnifying glass
C.) 2mL autosampler vials with Teflon caps
D.) GC/FID: HP 6890 or 7890A
E.) GC/MS: HP 6890/5973 or HP 7890A/5975C series
F.) Computer with Identidex Imprint Identification program.

IV. Procedure:

A.) Imprint Identification

1. Observe any imprint on tablet or capsule samples. Use a microscope or magnifying glass if necessary.
2. Record actual imprint, color, and shape of tablets or capsules in logbook.
3. On computer with Micromedex Healthcare Series, log onto the Identidex Imprint Identification page.
4. Enter the imprint code.
5. The program will access the database.
6. The identification, description and classification of the drug will appear. Read and verify that the

computer's description matches your sample's description (See example, last page).

7. Print out the results, record results in logbook and file with the sample paperwork.

B.) Chromatography by GC/FID and GC/MS

1. If capsule or tablet contains no identifiable imprint code (such as a picture) or if sample is in a powder form, the sample must be analyzed by GC/FID and GC/MS.
2. Place ¼ to ½ of tablet/capsule or 5 mg of powder sample into a 2 mL autosampler vial.
3. Add 9:1 Methylene chloride/NH_4OH.
4. Place on GC/FID autosampler and run with regular sequence (STD, BLK, Samples).
5. GC/FID conditions are as follows:

   Method: EXP.M
   Oven:
   Initial Temp: 245°C
   Initial Time: 0.00 min.
   Rate: 10°/min.
   Final Temp: 290°C
   Run Time: 10 min.
   Max. Temp: 325°C
   Equilibration Time: 0.5 min.
   Inlet:
   Mode: split (35:1)
   Initial Temp: 250°C
   Pressure: 24.99 psi
   Gas Type: Helium
   Column:
   Capillary: HP-1 30m x 320um
   Initial Flow: 3.3 mL/min.
   Detector:
   Temp: 300°C
   Hydrogen Flow: 30.0 mL/min.
   Air Flow: 400 mL/min.
   Makeup Gas: Helium

6. Obtain chromatographs. If sample contain amphetamine, methamphetamine, or any other phenethylamines, the instrument will detect a total ion peak with a retention time characteristic of that compound and

will generate a report with accompanying chromatograph.

7. Depending on results, analyst may want to rerun the sample using Petroleum Ether/NH_4OH.
8. Check concentration to determine if dilutions are needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates that the sample may have to be reinjected.
9. Amphetamines and Methamphetamines are early eluents so a different method will need to be run on the GC/MS that allows for a slower run time.
10. Place same sequence ran on the GC/FID on the GC/MS autosampler and run.
11. GC/MS conditions are as follows:
    Method: HYD.M
    Oven:
        Initial Temp: 130°C
        Initial Time: 2.50 min.
        Max. Temp: 325°C
        Equilibration Time: 0.50 min.
        Rate: 10°/min.
        Final Temp: 280°C
        Run Time: 30 min.
        Inlet:
        Mode: split (50:1)
        Initial Temp: 250°C
        Pressure: 31.65 psi
        Gas Type: Helium
        Column:
        Capillary: HP-1MS 30m x 0.530mm
        Max. Temp: 300°C
        Initial Flow: 1.0 mL/min.
12. If amphetamine/methamphetamine is present in sample, the instrument will detect a total ion peak at its characteristic retention time and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance.

V. Results:

A.) For the Identidex Imprint Identification procedure, record the identity of the sample in the logbook. Also, print out the results from the computer and file it with that sample number's paperwork.

B.) Record results of the GC/MS in logbook. Then transfer the results to Drug Lab Results sheet that came with the samples. Be sure to include date of analysis, results, the number of tests performed per sample, and signature.

C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.